IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the application of
Koichi Nishimura et al
Serial No. 09/926,824
Fled: December 26, 2001

Group Art Unit: 1711
Primary Examiner:
Thao T. Tran

For: RUBBER COMPOSITION FOR HOSE AND HOSE

DECLARATION UNDER 37 C.F.R. 1.132

Honorable Commissioner of Patents and Trademarks
Washington, D.C. 20231

Sir:

I, Shigeru Fujita, of c/o Research and Development Center, Zeon Corporation, 2-1, Yako 1-chome, Kawasaki-ku, Kawasaki-shi, Kanagawa, 210-9507 Japan, being duly sworn, declare and state:

THAT I am by profession a research chemist having had a major in organic synthetic chemistry and been awarded a master's degree from the post-graduate course of the Faculty of Industrial Chemistry, Kantogakuin University in March 1993.

THAT I have been employed since April 1993 by Zeon Corporation of 6-1, Marunouchi 2-chome, Chiyoda-ku, Tokyo, 100-0005 Japan and engaged in research on properties of polymers, especially hydrine rubber and hydrogenated nitrile-butadiene rubber in the Research & Development Center, Elastomer Laboratory of the same company.

THAT I am a co-inventor of the invention disclosed in the above-identified U.S. patent application (hereinafter referred to as "present invention") and hence I am fully familiar therewith.

THAT, I was intimately involved with the preparation of the above-identified U.S. patent application, and that all the examples in the above-identified U.S. patent application correspond exactly to experiments made for preparing the application and by knowledge are true; and that all statements made in association therewith in the above-identified U.S. patent application are made on information and belief and are believed to be true.

THAT, in order to show that the rubber composition for hose of the present invention provides unexpected and surprising results over Starmer U.S. Patent No. 4,048,261, the following experiments have been carried out under my supervision.

Comparative Experiments

Starmer '261 patent discloses a rubber composition comprising a diene/nitrile rubber, an epihalohydrin rubber and a crosslinking agent.

In the working examples of Starmer '261 patent, two types of diene/nitrile rubbers are specifically disclosed, which have the following compositions.

Diene/nitrile rubber A comprising 66% butadiene units, 32% acrylonitrile units and 2% vinyl benzyl chloride units.

Diene/nitrile rubber B comprising 53% butadiene units, 44% acrylonitrile units and 3% vinyl benzyl chloride units.

Among the working examples of Starmer '261 patent, Example II, Run No. 3, 7 and 8 appear relatively relevant to the present invention because the following rubber compositions are specifically used in these examples.

Rubber Composition (1) (Example II, Run No. 3 in Starmer '261 patent) comprising 50% by weight of 66% butadiene/32%

acrylonitrile/2% vinyl benzyl chloride copolymer rubber and 50% by weight of epichlorohydrin copolymer rubber, and 1.5% (based on the weight of the rubber ingredients) of a co-curing agent.

Rubber Composition (2) (Example II, Run No. 7 in Starmer '261 patent) comprising 25% by weight of 53% butadiene/44% acrylonitrile/3% vinyl benzyl chloride copolymer rubber and 75% by weight of epichlorohydrin copolymer rubber, and 1.5% (based on the weight of the rubber ingredients) of a co-curing agent.

Rubber Composition (3) (Example II, Run No. 8 in Starmer '261 patent) comprising 75% by weight of 53% butadiene/44% acrylonitrile/3% vinyl benzyl chloride copolymer rubber and 25% by weight of epichlorohydrin copolymer rubber, and 1.5% (based on the weight of the rubber ingredients) of a co-curing agent.

In the comparative experiments, three rubber compositions having the above-mentioned compositions similar to those disclosed in Example II, Run No. 3, 7 and 8 of Starmer '261 patent were prepared, and crosslinked rubber sheets and hoses were made therefrom and their resistance to fuel oil permeation and cold resistance were evaluated.

That is, rubber compositions were prepared from a nitrile rubber, an epihalohydrin rubber, a crosslinking agent and a crosslinking accelerator according to the recipes shown in Table I and by the same procedures as described in Example 1 of the above-identified U.S. patent application.

Each rubber composition was crosslinked to prepare a crosslinked rubber sheet having a thickness of 2 mm, and its properties were evaluated, by the same procedures as described in Example 1 of the above-identified U.S. patent application.

A cylindrical hose having a single layer structure was made from each rubber composition and crosslinked, and its cold resistance and fuel oil permeation resistance were evaluated, by the same procedures as described in Example 1 of the above-identified U.S. patent application.

The obtained results are shown in Comparative Examples 4, 5 and 6 in Table I, below.

For comparison, the results obtained in Examples 1 and 2 of the above-identified U.S. patent application are also shown in Table I, below.

Table I

Example No.	Examples		Comparative Examples		
	1	2	1	2	3
Composition (parts)					
Nitrile Rubber A1*[1] AN unit content: 53%	50	-	-	-	-
Nitrile Rubber A2*[2] AN unit content: 50%	-	60	-	-	-
Nitrile Rubber A4*[3] AN unit content: 32%	-	-	50	-	-
Nitrile Rubber A5*[4] AN unit content: 44%	-	-	-	25	75
Epihalohydrin Rubber B1*[5]	50	-	-	-	-
Epihalohydrin Rubber B2*[6]	-	40	50	75	25
Crosslinking Agent C_A1 for Nitrile Rubber: Sulfur	1	1	-	-	-
Crosslinking Accelerator*[7]	1	1	-	-	-
Acid Acceptor: Magnesium oxide*[8]	1.5	1.5	1.5	1.5	1.5
Crosslinking Agent C_B1*[9] for Epihalohydrin Rubber	2	2	-	-	-
Crosslinking Agent C_B2*[10] for Epihalohydrin Rubber	-	-	1.5	1.5	1.5
Properties of Crosslinked Rubber Sheet					
Permeation of fuel oil C ($g \cdot mm/m^2 \cdot day$)	195	205	430	180	312
Low-temp. impact brittle point (°C)	-28	-27	-31	-20	-21
Tensile strength (MPa)	15.2	15.6	14.3	12.9	16.8
Breaking elongation (%)	290	310	350	390	350
Hardness (JIS A)	69	71	66	69	71
Properties of Hose					
Permeation of fuel oil C (g/day)	0.25	0.29	0.83	0.31	0.48
Cold resistance*[11] at -30°C	A	A	A	A	A
at -35°C	A	A	A	B	B

Note,

*1 Nitrile Rubber A1: "Nipol DN002" available from Zeon Corporation, acrylonitrile content: 53%, ML_{1+4},100°C: 50

*2 Nitrile Rubber A2: "Nipol DN003" available from Zeon Corporation, acrylonitrile content: 50%, ML_{1+4},100°C: 78

*3 Nitrile Rubber A4: Copolymer of 32% acrylonitrile units, 66% butadiene units, and 2% vinyl benzyl chloride

*4 Nitrile Rubber A5: Copolymer of 44% acrylonitrile units, 53% butadiene units, and 3% vinyl benzyl chloride

*5 Epihalohydrin rubber B1: "Gechron 3100" available from Zeon Corporation, epichlorohydrin-ethylene oxide-allyl glycidyl ether terpolymer, ML_{1+4},100°C: 70

*6 Epihalohydrin rubber B2: "Gechron 1100" available from Zeon Corporation, epichlorohydrin-allyl glycidyl ether copolymer, ML_{1+4},100°C: 58

*7 Crosslinking accelerator: Dibenzothiazyl disulfide, "Nocceler DM" available from Ouchi Shinko Chem. Ind. Co., Ltd.

*8 Magnesium oxide: "Kyowa Mag 150" available from Kawaguchi Chem. Ind. Co., Ltd.

*9 Crosslinking agent C_B1: Ethylene thiourea, "Accel 22" available from Kawaguchi Chem. Ind. Co., Ltd.

*10 Crosslinking agent C_B2: 2,4,6-trimercapto-s-triazine, available from Sankyo Kasei Co., Ltd.

*11 Cold resistance: rating A: crack did not occur
rating B: crack occurred

Conclusion

As seen from the comparative experiments, the rubber compositions which are specifically disclosed in Example II, Run No. 3, 7 and 8 in Starmer '261 patent and have the following compositions gave hoses exhibiting poor resistance to permeation to fuel oil or poor cold resistance, as compared with rubber compositions of the present invention.

Rubber Composition (1) (Example II, Run No. 3 in Starmer

'261 patent) comprising 50% of 66% butadiene/32% acrylonitrile/2% vinyl benzyl chloride copolymer rubber and 50% of epichlorohydrin copolymer rubber, and 1.5% of a co-curing agent.

Rubber Composition (2) (Example II, Run No. 7 in Starmer '261 patent) comprising 25% by weight of 53% butadiene/44% acrylonitrile/3% vinyl benzyl chloride copolymer rubber and 75% by weight of epichlorohydrin copolymer rubber, and 1.5% of a co-curing agent.

Rubber Composition (3) (Example II, Run No. 8 in Starmer '261 patent) comprising 75% by weight of 53% butadiene/44% acrylonitrile/3% vinyl benzyl chloride copolymer rubber and 25% by weight of an epichlorohydrin copolymer rubber, and 1.5% of a co-curing agent.

I, the undersigned declarant, declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and; further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001, of Title 18, of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

This eighth day of August, 2003

Shigeru Fujita

Shigeru Fujita